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US EPA RECORDS CENTER REGION 5



513976

CERTIFIED MAIL
RETURN RECEIPT REQUESTED

July 8, 1988

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United States Environmental
Protection Agency, Region 5
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Commissioner
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Director, Solid and Hazardous
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Minnesota Pollution Control Agency
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President
Reilly Tar & Chemical Corporation
1510 Market Square Center
151 North Delaware
Indianapolis, Indiana 46204

RE: United States of America, et al. vs. Reilly Tar &
Chemical Corporation, et al.
File Civ. No. 4-80-469

Gentlemen and Sister Ashton:

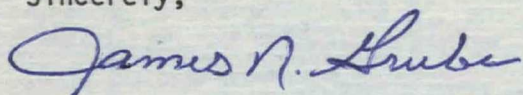
Pursuant to receipt of the agencies' May 26, 1988 approval letter relative to the City's submittal of the Soil Investigation Plan, issued in accordance with Section 11.1.1. of the Consent Decree - Remedial Action Plan in the referenced case, the City hereby submits modifications to the Plan as requested. To assist in the review of the modifications, the following comments are offered:

- Item I - Correction of name has been made on the title page.
- Item II - The "List of Figures" has been added to the "Table of Contents" element. Refer to page 2.
- Item III.1. - A time schedule has been developed using a bar chart. Refer to page 7A.
- Item III.2. - Data Quality Objectives have been incorporated in Section 5.1, page 11.
- Item III.3. - The Sampling Rationale now contains a reference to the location of the sampling. Refer to Section 3.5.1., page 7.

- Item IV.1. - Field duplicate frequency has been changed to 10%. Refer to Section 5, page 11.
- Item IV.2. - Criteria used to establish soil contamination is outlined in Section 5, page 11.
- Item V.1. - Decontamination methods now incorporate methanol and air drying. Refer to Section 6.4., page 16.
- Item V.2. - All samples will be analyzed for benzene extractable hydrocarbons per Section 6.4., page 16.
- Item VI.1. - Figure 7-1 has been removed and all other figures in Section 7 have been adjusted. Refer to page 18.
- Item VII.1. - Acceptance criteria for duplicates have been developed. Refer to Section 9.1.2.H.7., page 28 and Section 9.1.3.H.6., page 32.
- Item VII.2. - The typographical error in Section 9.1.1., page 25, has been corrected.
- Item VII.3. - Incorrect references to "Standard Methods for the Examination of Water and Wastewater" have been changed. Refer to Section 9.1.1., page 25 and Section 9.1.3., page 30.
- Item VIII - Calibration procedures for field instruments are included in Section 8.3., page 24 and Standard Operating Procedure (SOP) STS-01.
- Item IX - Data reduction calculations are included in Section 10.2, page 33.
- Item X - Preventative maintenance procedures for HNU equipment are provided in Section 13.3, page 40.A., and SOP STS-02.
- Item XI - Data completeness issues has been developed under Section 14.4, pages 43 and 43A.
- Item XII - Corrective action responsibilities and procedures are provided under Sections 15.2 and 15.3, page 44.

It is trusted the referenced corrections adequately address the comments/questions offered by the agencies.

Sincerely,



James N. Grube
Director of Public Works

JNG/cmr
attachment

QUALITY ASSURANCE PROJECT PLAN

QUALITY ASSURANCE PROJECT PLAN
NEAR SURFACE CONTAMINATION
CONSENT DECREE - REMEDIAL ACTION PLAN
SECTION 11

Prepared for
The City of St. Louis Park
St. Louis Park, MN 55416

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APPROVED BY: James N. Grube..... DATE: 10/27/87.....
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QUALITY ASSURANCE PROJECT PLAN

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3.3 Time Schedule

In accordance with the contents of the Remedial Action Plan (RAP), Section 11.1.2, the drilling activities will take place within 90 days of receipt of approval of the plan by the USEPA and MPCA, or within a different time frame upon mutual agreement of the Agencies, Reilly, and City. Within 60 days of completing installation of borings, the City shall submit to the USEPA and MPCA a report on the results of the borings.

Figure 3-2 presents the schedule currently planned for the installation and sampling of the borings. This schedule is subject to modification as the work progresses, and the City makes no commitments to meeting any of the schedule dates other than the completion/submittal requirements specified in the RAP.

3.4 Intended Data Usage

The qualitative descriptions of certain soil samples in this study shall be used to interpret whether samples are contaminated. The City shall coordinate an effort to notify the Parties to the CD-RAP owning property in the study area on which a release of hazardous substances resulting from operations at the Site has occurred or is occurring through the filing of affidavits with the Recorder of Deeds of Hennepin County pursuant to Minnesota Statutes 115B.16, Subdivision 2 (1984) within 180 days of completion of the borings. In addition, the City will submit a list of owners and locations of the properties on or under which a release of hazardous substances has occurred or continues to occur.

3.5 Sampling Network and Rationale

The Work Plan specifies the location of the initial 15 borings. These sampling locations have been defined utilizing the following three criteria:

1. Coverage of a variety of properties -- the study area constitutes approximately 300 acres of land in St. Louis Park. Soil borings have been proposed over 15 independently owned properties. Refer to Exhibit A of the Soil Investigation Plan for the locations of the borings.
2. Areal coverage maximized -- in addition to studying as many properties as is possible, the soil boring locations have been proposed to cover as large an area as possible.
3. Ease of access to the boring location -- in order to use the funds for this study in a cost effective manner reasonable access with the drilling equipment for each boring location has been sought.

3.6 Sample Matrices, Parameters and Frequency

Section 11.1 of the explicitly states that soils must be analysed for benzene extractables and/or Phenolics. Accordingly, for purposes of this Plan, the sample matrix shall simply be described as soil. No distinction of soil types is offered, however, during the course of the investigation all soil types encountered shall be reported.

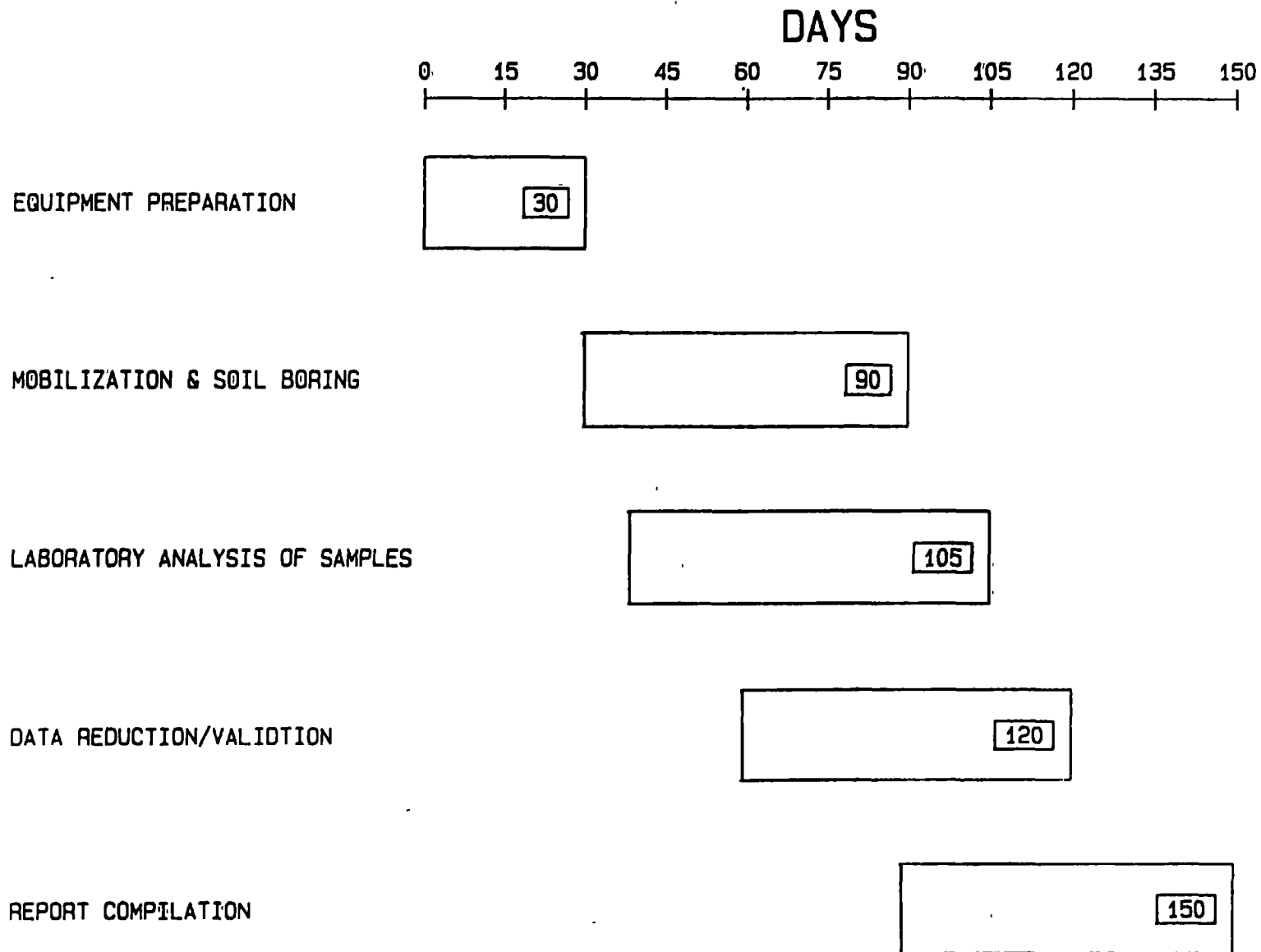


FIGURE 3-2
PRELIMINARY SCHEDULE FOR
SOIL BORING & SAMPLE ANALYSIS

QUALITY ASSURANCE PROJECT PLAN

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Each soil matrix retrieved for analysis shall be analyzed for benzene extractables and/or phenolics only. The frequency of sample retrieval/analysis, as established in the CD-RAP, is one time only. No repetition of sampling is contemplated unless the resampling constitutes one of the 15 to 25 borings and one of the 15 to 45 analyses.

5. QUALITY ASSURANCE OBJECTIVES

The principal objectives of this Plan pertain to the collection of data that are sufficient to determine the absence or presence of soil contamination above normal background levels for phenolics and benzene extractables. The CD-RAP defines Contamination as "PAH and Phenolics resulting from activities of Reilly at the (former coal tar refinery and wood treating) Site when found in the groundwater or the soil." The first criterion that will be used to define contamination is visual observation. This qualitative criteria is appropriate because the types of contaminants associated with Reilly's activities often produce a dark stain or discoloration of the soil. The presence of odors and organic vapors detected with the organic vapor detector/analyzer will supplement the visual descriptions of contamination. Pages 13 through 18 of the Site Management Plan provide greater detail relative to the definition of background levels, contamination, etc.

The quality of the data gathered in this project can be defined in terms of the following elements:

- a. **Completeness** - a sufficient number of successful (valid) measurements to characterize the concentrations of phenols and benzene extractables in St. Louis Park sampling sites.
- b. **Representativeness** - the extent to which reported analytical results truly depict the phenolic and benzene extractable concentrations in the sampled environment. Representativeness is optimized through proper selection of sampling sites, through proper sample preservation, and through prompt analysis.
- c. **Accuracy and Precision** - accurate and precise data will be achieved through the use of sampling and analytical procedures that minimize biases, through the use of standard procedures, through the meticulous calibration of analytical equipment and by implementing corrective action whenever measured accuracy and precision exceed pre-established limits. Accuracy and precision will be measured by the analysis of method spikes and duplicate samples.
- d. **Sensitivity** - determination of instrument sensitivity is accomplished by calibration using multiple concentrations of the analytes of interest. Once instrument sensitivity is demonstrated, analysis of duplicate spiked samples of deionized reagent water at a concentration of 1-5 times the instrument sensitivity is used to determine method sensitivity (i.e., method detection limit).
- e. **Comparability** - the extent to which comparisons among separate measurements will yield valid conclusions. Comparability among measurements in the SLP monitoring program will be achieved through the use of rigorous standard sampling and analytical procedures.
- f. **Traceability** - the extent to which results can be substantiated by hard-copy documentation. Traceability documentation exists in two forms: That which links final numerical results to authoritative measurement standards, and that which explicitly describes the history of each sample from collection to analysis.

The mechanisms that will be employed to achieve these quality goals are categorized as prevention, assessment and correction, as follows

- a. Prevention of defects in the quality through planning and design, documented instructions and procedures, and careful selection and training of skilled, qualified personnel.
- b. Quality assessment through a program of regular audits and inspections to supplement continual informal review.
- c. Permanent correction of conditions adverse to quality through a closed-loop corrective action system

The Quality Assurance objectives will include method blanks, field duplicates and matrix spikes. Precision, accuracy and completeness criteria are established for each parameter of interest. The specific criteria for each analysis and parameter are set forth in detail in the following sections:

<u>Objectives</u>	<u>Frequency</u>
Field Duplicates	10%
Method Blanks	5%
Matrix Spikes	5%

5.1 Data Quality Objectives

5.1.1 Accuracy

The required analyte recovery for both phenolic and benzene extracted spiked samples shall be a minimum of 50% and a maximum of 180%.

5.1.2 Precision

The required reproducibility on split sample analysis for both phenolics and benzene extractables shall be a relative percent difference no greater than 50%.

5.1.3 Sensitivity

Required detection limit for benzene extractables shall be 500 mg/kg, and 1 mg/kg for phenolics.

5.1.4 Completeness

90% of analytical data shall meet the above criteria for accuracy, precision and sensitivity.

4. Whenever samples are split with another laboratory, it is noted in the "Remarks" section. The note indicates with whom the samples are being split and is signed by both the sampler and recipient. If either party refuses a split sample, this will be noted and signed by both parties. The person relinquishing the samples to the facility or agency should request the signature of a representative of the appropriate party, acknowledging receipt of these samples. If a representative is unavailable or refuses to sign, this is noted in the "Remarks" space. When appropriate, as in the case where the representative is unavailable, the custody record should contain a statement that the samples were delivered to the designated location at the designated time.

6.3.3 Field Forms

In addition to sample labels and chain-of-custody forms, a bound field notebook will be maintained by the sample team leader to provide a daily record of significant events. All entries will be signed and dated. All members of the sampling team will use this notebook. The notebook will be kept as a permanent record.

6.4. Sampling Procedures

For this study, background levels of benzene extractable hydrocarbons will be determined by testing a maximum of 10 samples classified by the sense of smell as "clean". The City's representative will be responsible for designating at least one sample retrieved from each soil boring for laboratory analysis for benzene extractable hydrocarbons. Accordingly, at least 25 samples will be analyzed during the initial round of sampling. In the event up to 10 additional soil borings are undertaken in a follow-up round, at least one sample will be returned from each boring, up to a total of 20 samples, and all samples will be analyzed for benzene extractable hydrocarbons.

Soil samples, weighing, at least 200 grams will be obtained using the split-barrel sampling procedure in general conformance with ASTM Specification D-1586-84. The geologist will take possession of the split-barrel sampler immediately upon its emergence from the borehole, prepare all soil samples for laboratory analysis, and classify the samples in m.l. wide mouth clear glass sample containers fitted with aluminum foil lined caps. All samples shall be packed, cooled to a temperature less than 4 C and shipped to the analytical laboratory on the same day.

A new pair of disposable latex gloves will be used for each sampling site. Between sites and between each use of the split-barrel sampler, sampling equipment will be steam cleaned and rinsed with methanol, hexane, methanol, air dried in a contaminant free area and then rinsed with deionized water prior to reuse.

6.5. Field Measurement Equipment

All field measurement equipment will be controlled in accordance with manufacturer's specifications to ensure that measurements obtained are accurate and defensible. Specific field measurement equipment shall include an HNU Photoionization Detector.

6.6. Duplicate Samples

Duplicate samples will be collected by splitting the sample longitudinally with a stainless steel knife, and each half will be placed in separate sample jars.

7. SAMPLE CUSTODY

Interpoll operates under a formal quality control program. The Chain-of-Custody (Figure 6-2) contains two major elements: The field sampling, and the laboratory custody. Section 6.3 discusses the field sampling aspects. This section covers quality related activities applicable to the St. Louis Park Soil Study from the receipt of samples at the laboratory through the issuance of validated analytical data and the storage of data in the final evidence file.

7.1 Chain-of-Custody

When samples are received into the laboratory, the Sample Custodian will verify their integrity as they are unpacked and will explicitly state in the log-in records whether the sample is received intact or broken and whether the sample is appropriately identified. If the integrity requirements are met, or when any discrepancies are resolved, Interpoll assigns the sample a laboratory control number, stores the samples in a refrigerator and enters the pertinent information into the sample log.

7.2 Recordkeeping

In addition to sample chain-of-custody, the laboratory will maintain the necessary documentation to reconstruct the entire process of sample preparation through analysis and report generation. This documentation is found in logbooks, data packages.

The data package contains only data pertinent to the individual project. This package is filed alphabetically by project and date and includes the following records:

- a. Forms Distribution Summary - a form which lists the contents of the Data Package and routes the data review process (Figure 7-1).
- b. Out-of-Control Events Log - a form which describes any out-of-control events which may affect the quality of data to be reported and explains the causes and corrective actions taken (Figure 7-2).
- c. Analytical method QC Checklist - on this sheet one records pertinent information from duplicate and spiked samples, method blanks and performance standards (Figures 7-3 and 7-4).

In addition to these forms, a Data Package will contain other pertinent information, such as daily instrument calibration, check standard results, chromatographic charts, computer printouts, references to other logbook entries and correspondence.

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Forms Distribution Summary

Due Date _____

CLIENT: _____

JOB: _____

CLIENT NO: _____

P.O. NO: _____

PHONE: _____

PROJECT MGR: _____

CONTACT: _____

DATE: _____

LABORATORY REPORT #: _____

SAMPLES COLLECTED: _____

SAMPLES RECEIVED: _____

(DRS = "Data Reporting Sheet")

(DS = "Data Sheet")

Invoicing

Signature

Report
Routing

PL
Lab Mgr
Ino Mgr
Org Mgr

Inorganic

____ LCI-17, Inorganic Area DRS
(Fill out or add to corresponding
form LCI-19)

Metals

____ LCI-04R, Metals DRS
____ LCI-07R, E.P. Toxicity DRS
(Also fill out form LCI-37)
____ LCI-08R(3), EPA Waste Oil Profile DRS
____ LCI-09R(1), ASTM Leach DRS
____ LCI-15R, Mineral Ash DRS
____ LCI-34(4), MPCA Waste Profile DRS
____ LCI-36(1), Incineration Param. DRS

Fuel

____ LCI-02R, Fuel DRS
____ LCI-08R(1), EPA Waste Oil Profile DRS
____ LCI-20, Fusion Temperature of Ash DRS
____ LCI-34(1), MPCA Waste Profile DRS
____ LCI-36(2), Incineration Param. DRS

Ion Chromatography

____ LCI-08R(2), EPA Waste Oil Profile DRS
____ LCI-09R(2), ASTM Leach DRS
____ LCI-21, Ion Chromatography DRS
____ LCI-34(2), MPCA Waste Profile DRS
____ LCI-36(3), Incineration Param. DRS

Organic

____ LCI-05, VOA DRS
____ LCI-06, PAH DRS
____ LCI-18, Phenols DRS
____ LCI-28, PCB DRS
____ LCI-29, Pesticide/Herbicide DRS
____ LCI-34(3), MPCA Waste Profile DRS
____ LCI-35(1-4), EPA Method 625 DRS

GC/MS

____ LCI-13, Request for GC/MS Analysis

Acid Rain

____ LAR-01, Wet Deposition
Metal Analysis DS
____ LAR-02, Dry Deposition
Ammonium IC Analysis
____ LAR-03, Wet Deposition
Cation Chromatography Sheet
____ LAR-04, Wet Deposition
Sample Prep, pH, Conductivity
____ LAR-05, IC Anion Analysis

Particle Sizing

____ LCI-25, Request for Particle Size
Analysis
____ LCI-26, Particle Size Distribution
Analysis
____ LCI-27, Combining Particle Size Results

Comments:

Out of Control Events Log

Analyte _____ Method _____

Date _____ Units of Measurement _____

Nature of Out of Control Situation: _____

Discussion: _____

Action Taken: _____

Analyst _____ Reviewed by _____ QCC

Dept. Mgr. _____ QAM

Lab Director

Analytical Method QC Checklist

Project Name: _____ Date: _____

Category: _____ Analyst: _____

Matrix: _____ Batch Numbers: _____

Prep Method #: _____

Analytical Method #: _____

Total Number of Samples Analyzed: _____

1. Values for all instrument blanks below detection limit: Yes _____ No _____

2. Reference standard analysis: Source _____

<u>Analyte</u>	<u>Theoretical Value</u>	<u>Observed Value</u>	<u>Percent Recovery</u>
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____

3. Matrix Spikes

Number of spiked samples analyzed: _____ Average % Recovery: _____

Range of % Recovery: _____

Recovery of each spike within control limits: Yes _____ No _____

4. Duplicates

Number of duplicate samples analyzed: _____

Average Relative Percent Difference: _____

Range of Relative Percent Difference: _____

Precision for each set of duplicates within control limits: Yes _____ No _____

5. Method Blank

Number of method blanks analyzed: _____

All below detection limit: Yes _____ No _____

6. Calibration verified every _____ samples.

Comments: _____

Interpol: Laboratories

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Category: _____
Matrix: _____
Prep Method #: _____

Date: _____
Analyst: _____

[illegible][illegible]

DATA PROCESSED BY: _____ ON: _____

QC DATA REPORT

8. CALIBRATION PROCEDURES

8.1 Analysis of Phenolics

Prior to use of the method for analysis of phenols, a five-point response factor calibration curve is established showing the linear range of the analysis. Daily response factors for phenol are compared to the initial calibration curve. If the daily response factors are within +10 percent of the corresponding calibration curve value, the analysis may proceed. If, for any analyte, the daily response factor is not within +10 percent of the corresponding calibration curve value, a five-point calibration curve must be repeated prior to the analysis of samples.

8.2 Analysis of Benzene Extractables

Prior to use of the method of analysis of benzene extractables, an analytical balance, which is semi-annually calibrated under a service contract, is checked with class S weights to assure proper operation.

8.3 Field Measurement Equipment

8.3.1 HNU Photoionization Detector

The photoionization detector must be calibrated each day prior to field use. A calibration gas will be taken into the field to perform this routine calibration check. The procedure for the calibration of the HNU photoionization detector is listed in Standard Operating Procedures (SOP) STS-01, Appendix A to this Quality Assurance Project Plan.

8.3.2 Biosensor II Combustible Gas Indicator

The combustible gas indicator must be calibrated each week. The procedure for calibrating the Biosensor II combustible gas indicator is listed in SOP STS-02, Appendix A to this Quality Assurance Project Plan.

9. ANALYTICAL PROCEDURES

9.1 Analysis of Phenolics and Benzene Extractables

9.1.1 Summary

Previously, the level of phenolic material in soil samples was measured on a 3 to 5 gram soil sample using the "Distillation Chloroform Extraction Procedure" contained on page 558 of "Standard Methods for the Examination of Water and Wastewater, 16th Edition." An April 20, 1970 memorandum to the Minnesota Pollution Control Agency from the Minnesota Department of Health concluded that this general method of phenolic determination was adequate to measure phenolics discharged in creosoting wastes from the Site, based on a limited amount of comparative testing carried out in the Health Department's laboratory. The detection limit for this phenolic analysis procedure was approximately 0.2 mg/kg (wet weight) when applied to the soil samples. Benzene extractable material was measured by extracting a 20 gram soil sample with benzene in a Soxhlet extraction apparatus for four hours and measuring the total weight of material extracted. Except for the use of benzene as the solvent, the analytical procedure used to measure the concentration of extractable material was the same as the Soxhlet extraction procedure given on page 412 of "Standard Methods for the Examination of Waste and Wastewaters 3th Edition." Extracted material was reported in milligrams of extracted material per kilogram of sample. The detection limit for the benzene extractable analyses was 50 mg/kg (wet weight).

To measure the concentration of phenolic material and benzene extractable material, a 100 gram sub-sample was taken from each soil sample and the sub-sample was quartered to obtain a 25 gram sample. Twenty grams of the quartered sub-sample were used in the analysis for benzene extractable material and 3 to 5 grams of the quartered sub-sample are used in the analysis of phenolic material.

To obtain the moisture content and, therefore, the dry weight of the soil sample, a quarter of the sub-sample was oven dried at 105 C to a constant weight. The loss of hydrocarbons by oven drying the samples at 105 C was defined by comparing the moisture content obtained by oven drying with the moisture content obtained by air drying.

9.1.2 Benzene Extractables

A. SCOPE AND APPLICATION

1. This method is used to recover benzene extractables by chemically drying a wet sludge sample and then extracting via the Soxhlet apparatus.

11. Rinse flask and cotton with solvent.
12. Evaporate the solvent by immersing the lower half of the flask in water of 90 C. A solvent blank should accompany each set of samples.
13. When the flask appears dry, remove. To remove solvent vapor, sweep out the flask for 15 seconds with air by inserting a glass tube that is connected to a vacuum source. Immediately remove the flask from the heat source and wipe the outside to remove excess moisture and fingerprints.
14. Cool the boiling flask in a desiccator for 30 minutes and weigh.
15. Calculate benzene extractables as a percentage of the total dry solids. Generally:

$$\% \text{ of oil \& grease} = \frac{\text{gain in weight of flask, g} \times 100}{\text{wt. of wet solids, g} \times \text{dry solids fraction}}$$

H. QUALITY CONTROL

1. Before processing any samples, the analyst should demonstrate through the analysis of Type II water method blank that all glassware is free of organic contamination; if there is a change in reagents, a method blank should be processed as a safeguard against reagent contamination. The blank sample should be carried through all stages of the sample preparation and measurement.
2. Standard quality assurance practices should be used with this method. Laboratory duplicates should be analyzed to validate the precision of the analysis. Fortified samples should be carried through all stages of sample preparation and measurement; they should be analyzed to validate the sensitivity and accuracy of the analysis.
3. Comprehensive quality control procedures are specified for each target compound in the referring analytical method.
4. All quality control data should be maintained and available for easy reference or inspection.
5. Employ a minimum of one blank per sample batch to determine if contamination has occurred.
6. Verify calibration with an independently prepared check standard every 15 samples.
7. Run one spike duplicate sample for every 10 samples. A duplicate sample is a sample brought through the whole sample preparation and analytical process (relative percent difference of greater than 50% is out of control).

I. METHOD PERFORMANCE

1. No data provided.

J. REFERENCES

1. EPA Test Methods for Evaluating Solid Waste, SW-846, 3rd ed., Method 9071 (1986).
2. Blum, K.A. and M.J. Taras, "Determination of Emulsifying Oil in Industrial Wastewater", JWPCF Research Suppl., 40, R404 (1968).
3. Standard Methods for the Examination of Water and Wastewater, 16th ed., page 499.

9.1.3. Phenolics - EPA Method 9065 I

A. SCOPE AND APPLICATION

1. This method is applicable to the analysis of sediments and soils.
2. The method is capable of measuring phenolic materials at the 0.2 mg/L level when the colored end product is extracted and concentrated in a solvent phase using phenol as a standard.
3. The method is capable of measuring phenolic materials that contain more than 50 ug/L in the aqueous phase (without solvent extraction) using phenol as a standard.
4. It is not possible to use this method to differentiate between different kinds of phenols.

B. SUMMARY OF METHOD

1. Phenolic materials react with 4-aminoantipyrine in the presence of potassium ferricyanide at a pH of 10 to form a stable reddish-brown antipyrine dye. The amount of color produced is a function of concentration of phenolic material.

C. INTERFERENCES

1. Preliminary distillation is required to remove interfering materials.
2. Color response of phenolic materials with 4-aminoantipyrine is not the same for all compounds. Because phenolic-type wastes usually contain a variety of phenols, it is not possible to duplicate a mixture of phenols to be used as a standard. For this reason, phenol has been selected as a standard and any color produced by the reaction of other phenolic compounds is reported as phenol. This value will represent the minimum concentration of phenolic compounds present in the sample.
3. Interferences from sulfur compounds are eliminated by acidifying the sample to a pH <4 with H₂SO₄ and aerating briefly by stirring.

- f. After 3 minutes, extract with 25 mL of chloroform (E.9.). Shake the separatory funnel at least 10 times, let CHCl_3 settle, shake again 10 times and let chloroform settle again.
 - g. Filter chloroform extract through filter paper. Do not add more chloroform.
 - h. Read the absorbance of the samples and standards against the blank at 460 nm.
3. Calculation:
- a. Prepare a standard curve by plotting the absorbance values of standards versus the corresponding phenol concentrations.
 - b. Obtain concentration value of sample directly from standard curve.

H. QUALITY CONTROL

- 1. All quality control data should be maintained and available for easy reference or inspection.
- 2. Calibration curves must be composed of a minimum of a blank and three standards. A calibration curve should be made for every hour of continuous sample analysis.
- 3. Dilute samples if they are more concentrated than the highest standard or if they fall on the plateau of a calibration curve.
- 4. Employ a minimum of one blank per sample batch to determine if contamination has occurred.
- 5. Verify calibration with an independently prepared check standard every 15 samples.
- 6. Run one spike duplicate sample for every 10 samples. A duplicate sample is a sample brought through the whole sample preparation and analytical process (relative percent difference of greater than 50% is out of control).

I. REFERENCES

- 1. EPA Test Methods for Evaluating Solid Waste, SW-846, 3rd ed., Method 9065 (1986).
- 2. Annual Book of ASTM Standards, Part 31, "Water", Standard D17830-70, p. 553 (1976).
- 3. Standard Methods for the Examination of Water and Wastewater, 16th ed., page 558

10. DATA REDUCTION, VALIDATION AND REPORTING

10.1 Data Reduction and Validation

All data will be subjected to a rigorous review process before being reported. All data forms must be dated, signed and completely filled out in ink by the preparer. Notes will be made if information requested is non-applicable for the specific analysis. Each data sheet will be checked, signed, dated approved by someone other than the preparer.

Out-of-control events or potential out-of-control events are noted on an Out-of-Control Events form. This form is part of the Data Package and will be completed upon data approval. If no out-of-control event does occur during analysis (for instance, a spike recovery falls outside the expected range), the analyst will describe the event, the investigative and corrective action taken and the cause of the event on this form, and will notify the Quality Control Coordinator (QCC).

After an analyst completes a Data Package, it is given to the Supervisor for review. The Supervisor reviews the entire Data Package for completeness, discrepancies and errors and writes comments, when necessary, on the back of the Data Approval Form. If the Supervisor disapproves the Data Package, it is given back to the analyst for correction. If it is approved, the Supervisor passes it along to the QCC.

The QCC then reviews the Data Package with extra emphasis on the acceptability of quality control data. If the QCC disapproves the Data Package, it is re-routed to the Supervisor for corrective action. If the QCC approves it, it is sent to the Laboratory Manager, Supervisors and Quality Control Coordinator for their approval and signatures

10.2 Calculations

Concentration of Benzene Extractables = $\frac{\text{gain in weight of flask}}{\text{weight of sample}}$

Concentration of Phenolics = $\frac{\text{concentration from standard}}{\text{curve} \times \text{dilution factor}}$

Dilution Factor = $\frac{500}{\text{g of sample}}$

10.3 Turnaround

In accordance with Section 11.1.3 of the CD-RAP the City will report the results of the borings and laboratory analyses within 60 days of completion of the borings.

10.4 Analytical Results Report

Each analytical results report will contain the following:

- a. Field identification designation.
- b. Interpoll laboratory sample number.
- c. Analytical results (mg/kg) in terms of phenolics.

In addition, the City will make the Data Package, described in Section 7.2., available to the Agencies if so notified.

The analytical results report will be validated and signed by the Laboratory Manager.

13. PREVENTIVE MAINTENANCE

Since instrumental methods of analysis require properly maintained and calibrated equipment, the operation and maintenance of modern analytical instrumentation is of primary importance in the production of acceptable data. In order to provide this data, STS and Interpoll subscribe to the following programs.

- a. Maintenance agreements/service contracts with instrument manufacturers.
- b. Laboratory preventive maintenance program.

13.1 Service Contracts

Analytical equipment utilized in Interpoll laboratory personnel for this project are covered by maintenance agreements. These maintenance agreements provide for both periodic "preventive" service calls as well as the non-routine or emergency calls.

13.2 Instrument Logbooks

Individual instrument logbooks are maintained for each piece of equipment and located near the instrument. General information contained in the logbooks include:

- a. Inventory information: Equipment name, model number, serial number, manufacturer, date of acquisition, original cost.
- b. Service tasks and intervals: Cleaning, calibration, operation based on the manufacturer's recommended schedule, and previous laboratory experience.
- c. Service record: Date of breakdown, date of return to service, down time, problems, repairs, cost of repairs, who performed the repairs, parts required, etc.
- d. Calibration/performance checks.
- e. Daily operational notes.

Analysts are referred to manufacturer's operating manuals for specific procedures to be followed in the operation and/or maintenance of the individual instrument.

Laboratory preventive maintenance includes any tasks that can be performed in-house, i.e., systematic cleaning of component parts as recommended in the instrument manual. If problems cannot be corrected by laboratory personnel, the instrument service representative is contacted and a service call requested to correct the problem.

13.3 Field Measurement Equipment

The HNU photoionization detector used by STS in the field analysis of soil samples shall be maintained in accordance with the requirements of the manufacturer and as outlined in SOP STS-02, Appendix A to this Quality Assurance Project Plan.

14.2.3 Limits

Both upper and lower warning limits and upper and lower control limits are established to aid in interpreting a suspicious or an out-of-control event. Warning limits express a narrower confidence interval and are used to warn the analyst or supervisor of possible system inconsistencies or failures, before an out-of-control event occurs. Control limits express the out limits of expected method variability.

14.3 Suspicious/Out-of-Control Events

Graphing and connected successive data points on control charts enables the laboratory to detect many types of suspicious and out-of-control situation. These events can be caught by monitoring for the following: Outliers (suspicious and out-of-control), runs (suspicious), trends (suspicious) and periodicity (suspicious).

14.3.1 Outliers

There are two types of outliers: Any particular point that falls outside the control limits or any point that falls outside the warning limits. A point that falls outside the control limits is classified as an out-of-control event; a point that falls outside the warning limits is classified as a suspicious event.

14.3.2 Runs

A run is defined as a series of points that line up on one side of the central line (the mean). Any run that has a length of seven points is indicative of a potential abnormality in the process, a suspicious event. A run can suggest several potential problems such as elevated contamination or incorrect dilutions of standards.

14.3.3 Trends

A Trend is defined as a series of points that are marked by an unbroken rise or fall. Any trend with a length of five points is classified as a suspicious event. A trend may indicate a change in instrument sensitivity due to a dirty source or injection port or standard degradation, to name a few.

14.3.4 Periodicity

Periodicity is a term used to describe a recurring pattern of change over equal intervals. This occurrence may be of any length or amplitude; thus, careful observation of the control chart is necessary.

14.4 Acceptance Criteria

14.4.1 Accuracy

The required analyte recovery for both phenolic and benzene extracted spiked samples shall be a minimum of 50% and a maximum of 180%.

14.4.2 Precision

The required reproducibility of split sample analysis for both phenolic and benzene extractables shall be a relative percent difference no greater than 50%.

14.4.3 Sensitivity

Required detection limit for benzene extractables shall be 500 mg/kg, and 1 mg/kg for phenolics.

14.4.4 Completeness

90% of analytical data shall meet the above criteria for accuracy, precision and sensitivity.

15. CORRECTIVE ACTION

Corrective actions are required whenever an out-of-control event or potential out-of-control event is noted. The investigative action taken is somewhat dependent on the analysis and the event.

Generally, out-of-control events or potential out-of-control events are noted on an Out-of-control Events Form (Figure 15-1). This form is part of the data package and, thus, must be completed prior to data approval. If an out-of-control event does occur during analysis (for instance, a surrogate recovery falls outside the expected range), the analyst must describe on this form: The event, and notify the Laboratory Quality Control Coordinator (QCC). In some cases, investigation of an out-of-control event will reveal no problems. If an out-of-control event is discovered during data package review, the QCC notifies the supervisor for corrective action.

15.1.1 Matrix Spikes

Interpoll will use phenol spiked into a sample of soil collected in the field. The spiking levels will be two or three times the amount observed in the sample.

If the matrix spike criteria are not met, the matrix spike analysis will be repeated. If the subsequent matrix spike analysis meets the criteria, the data will be considered valid. Matrix spike recoveries will be used in assessing quality assurance/quality control for Interpoll's analytical work.

15.1.2 Duplicate Analysis

Interpoll will run duplicate analysis on 5% of submitted samples for both phenolics and benzene extractables.

15.2 Project Specific Out of Control Criteria

15.2.1 Accuracy

The required analyte recovery for both phenolic and benzene extracted spiked samples shall be a minimum of 50% and a maximum of 180%.

15.2.2 Precision

The required reproducibility on split sample analysis for both phenolics and benzene extractables shall be a relative percent difference no greater than 50%.

15.3 Accuracy and Precision Review

Accuracy and Precision data will be reviewed by the Quality Control Coordinator. If an out of control situation exists, this will be brought to the attention of the analyst and the Inorganic Chemistry Department Manager. After problem resolution, sign-offs are required by the Analyst, the QCC, the Department Manager, the Division Quality Control Manager and the Laboratory Director.

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Out of Control Events Log

Analyte _____ Method _____

Date _____ Units of Measurement _____

Nature of Out of Control Situation: _____

Discussion: _____

Action Taken: _____

Analyst _____ Reviewed by _____ QOC

Dept. Mgr. _____ QAM

Lab Director

APPENDIX A
STANDARD OPERATING PROCEDURES

INDEX OF STANDARD OPERATING PROCEDURES

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STANDARD OPERATING PROCEDURE
AIR MONITORING EQUIPMENT CALIBRATION

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INTRODUCTION

All monitoring instruments must be calibrated and maintained periodically. The limitations and possible sources of errors for the instrument must be understood by the operator. It is important that the operator ensures that the instrument responds properly to the substances it was designed to monitor. Below is the calibration and maintenance procedures for the HNU® photoionization detector and Biosensor®II combustible gas indicator.

HNU® Photoionization Detector

The photoionization detector must be calibrated each day prior to field use. A calibration gas will be taken into the field to perform this routine calibration check. The procedure for the calibration of a HNU® photoionization detector is listed below.

1. Attach the probe to the read-out unit. Match the alignment key, then turn the connector clockwise until a distinct locking is felt.
2. Turn the FUNCTION switch to the battery check position. Check to ensure that the indicator reads within or beyond the green battery arc on the scale plate. If the indicator is below the green arc, or if the red LED comes on, the battery must be charged prior to using the instrument.
3. To calibrate the zero of the instrument, turn the FUNCTION switch to the STANDBY position and rotate the ZERO POTENTIOMETER until the meter reads zero. Wait 15-20 seconds to ensure that the zero adjustment is stable. If not, rezero the instrument.
4. Check to see that the SPAN POTENTIOMETER is set at the appropriate setting for the probe being used.

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AIR MONITORING EQUIPMENT CALIBRATION

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5. Set the FUNCTION switch to the desired ppm range.
6. Listen for the fan operation to verify fan function.
7. Connect a sampling hose to the regulator outlet and the other end to the sampling probe of the HNU®.
8. Crack open the regulator valve to calibration gas.
9. Take reading after 5-10 seconds.
10. If the reading deviates $\pm 15\%$ from the concentration of the calibration gas, the instrument requires maintenance.
11. Results of calibration should be recorded in the logbook.
12. Correlate calibration gas valve to expected on-site contaminant.

NOTE: This instrument's operation may be verified periodically in the field with an organic point source such as a "magic marker."

Recommended maintenance for the HNU® is listed below:

<u>Function</u>	<u>Frequency</u>
o Wipe down read-out unit.	After each use.
o Clean UV light source window.	Every month.
o Clean ionization chamber.	Every month.
o Recharge battery.	Daily or as use dictates.

Biosensor®II Combustible Gas Indicator

The combustible gas indicator must be calibrated each week. The procedure for calibrating the combustible gas indicator (Biosensor®II) is listed below:

1. Attach the 0.5 liter per minute fixed flow rate regulator to the calibration gas cylinder.
2. Attach a sample line from the regulator to the balloon inlet. Attach another sample line from the balloon outlet to the sample draw intake on the instrument.

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AIR MONITORING EQUIPMENT CALIBRATION

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3. Fill the balloon with calibration gas and allow the sampling pump to draw it over the sensors. DO NOT OVERINFLATE THE BALLOON! Feed more gas into the balloon as needed to keep it partially inflated.
4. Wait for the readings to stabilize. Then, using a small jeweler's screwdriver, adjust the "gas span" potentiometer to obtain a steady reading which corresponds to the calibration gas concentration that is printed on the label of the calibration gas cylinder (normally 50 percent of the Lower Explosive Limit (LEL)).
5. Remove calibration lines.
6. Let the instrument run for one full minute to flush any excess calibration gas and check readings. The combustible sensor should now be reading zero percent LEL, (± .001 percent LEL) in fresh air. Repeat calibration procedures if necessary.
7. Results of the calibration must be recorded in the logbook.
8. Correlate the calibration gas valve to expected on-site explosive contaminants.

The Biosensor®II uses a 2 volt lead gel cell battery. This battery should be charged daily or as use dictates. The battery cannot be overcharged.

Colormetric Tubes

Colormetric tubes do not require calibration as they are single use items calibrated by the manufacturer through his QA procedures. The air pumping mechanism must be inspected and tested before each use to assure that the correct amount of air is passing through the colormetric tube. The air flow test consists of metering air flow with a manufacturer supplied calibration meter.

STANDARD OPERATING PROCEDURE
NHN PHOTOIONIZATION DETECTOR MAINTENANCE

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5.1 INTRODUCTION

Maintenance of the analyzer consists of cleaning the lamp and ion chamber, replacement of the lamp or other component parts or subassemblies.

WARNING: Turn the function switch on the control panel to the OFF position before any disassembly. Otherwise, high voltage of 1200 V DC will be present.

WARNING: Use great care when operating the analyzer with the readout assembly outside its case due to the presence of 1200 V DC.

WARNING: Do not look at the light source from any closer than 6 inches with unprotected eyes. Observe only briefly. Continued exposure to ultraviolet energy generated by the light source can be harmful to eyesight.

CAUTION: Do not interchange lamps of different eV ratings in a probe. Amplifier and components are selected for a specific eV lamp. A probe with the wrong lamp will not operate properly.

5.2 UV LAMP AND ION CHAMBER CLEANING

During periods of operation of the analyzer, dust or other foreign matter could be drawn into the probe forming deposits on the surface of the UV lamp or in the ion chamber. This condition is indicated by meter readings that are low, erratic, unstable, non-repeatable, or drifting, or show apparent moisture sensitivity. These deposits interfere with the ionization process and cause erroneous readings. Check for this condition monthly or as required. Cleaning can be accomplished as follows:

- a. Disassemble the probe and remove the lamp and ion chamber (see Section 5.5). Exercise great care in doing so to prevent inadvertent damage to these components.
- b. First check the lamp window for fouling by looking at the surface at an incident angle. Any deposits, films or discoloration may interfere with the ionization process. Clean the window as follows:

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NHN PHOTOIONIZATION DETECTOR MAINTENANCE

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1) 9.5 and 10.2 eV lamps

- a) First clean by rubbing gently with lens tissue dipped in a detergent solution.
- b) If this does not remove deposit, apply a small amount of HNU cleaning compound (PA101534) directly onto the lens of the lamp and spread evenly over surface with a non-abrasive tissue (e.g. Kim-Wipe) or a lens tissue.
- c) Wipe off compound with a new tissue.
- d) Rinse with warm water (about 80 degrees F) or damp tissue to remove all traces of grit or oils and any static charge that may have built up on the lens. Dry with new tissue.
- e) Reinstall lamp in detector and check analyzer operation.
- f) If performance is still not satisfactory replace the lamp. See Section 5.3 and Section 6.

2) 11.7 eV lamp

- a) Clean by putting a freon or chlorinated organic solvent on a tissue and rubbing gently.
- b) DO NOT CLEAN THIS LAMP WITH WATER OR ANY WATER MISCIBLE SOLVENTS (methanol or acetone). It will damage the lamp.
- c) DO NOT USE THE CLEANING COMPOUND used for the 9.5 and 10.2 eV lamps under any circumstances on the 11.7 eV lamp.

c. Then inspect the ion chamber for dust or particulate deposits. If such matter is present, the chamber can be cleaned by removing the outer Teflon ring, and the four screws holding the retaining ring. Carefully move the retaining ring aside (NOTE: this is soldered) and remove the screen. A tissue or cotton swab, dry or wetted with methanol, can be used to clean off any stubborn deposits. The assembly can also be gently swirled in methanol and dried gently at 50-60 degrees C for approximately a half hour. No liquid must be present at reassembly as this would affect the performance. Do not clean the ion chamber with the HNU cleaning compound cited above in para. b.1)b).

d. Reassemble the probe and check analyzer operation.

e. If performance is still not satisfactory replace the lamp. See Section 5.3.

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NHN PHOTOIONIZATION DETECTOR MAINTENANCE

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5.3 LAMP REPLACEMENT

To replace the lamp, disassemble the probe, remove the old lamp, install a new one of the same eV rating and reassemble.

WARNING

Turn the function switch on the control panel to the OFF position before disassembly. Otherwise, high voltage of 1200 V DC will be present.

CAUTION

Do not exchange lamps of different eV ratings in a probe. Amplifier and components are selected for a specific eV lamp. A probe with the wrong lamp will not operate properly.

Set the SPAN pot to 9.8 for the 10.2 eV lamp. Remove the readout assembly case (see Section 5.6). Locate the gain control potentiometer, R48, on the power supply board as shown on Figure 6-1. Recalibrate the analyzer adjusting this potentiometer, R48, with a small screwdriver to obtain the specified ppm reading, leaving the SPAN pot set at 9.8.

For the 9.5 and 11.7 eV lamps see the Application Data Sheet or calibrations memo for the proper span pot settings and readings.

WARNING..

Use great care when operating the analyzer with the readout assembly outside its case due to the presence of 1200 V DC.

When calibration is accomplished, turn the analyzer OFF and replace the readout assembly in its case.

Adjustment of R48 potentiometer is used only when a new lamp is installed. At all other times adjustment is accomplished using the SPAN control potentiometer.

If calibration cannot be achieved, see Section 6, Troubleshooting.

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NHN PHOTOIONIZATION DETECTOR MAINTENANCE

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SECTION 5 cont.

5.4 LAMP SIZE CHANGE

If different applications for the analyzer would require different size lamps, separate probes, each with its own eV lamp, must be used. A single readout assembly will serve for any of the probes. A change in probe will require resetting of the zero control and the span pot. Calibration should be checked to verify proper operation.

5.5 PROBE DISASSEMBLY/ASSEMBLY

WARNING

Turn the function switch on the control panel to the off position before disassembly. Otherwise high voltage of 1200 V DC will be present.

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SECTION 5.5, PROBE DISASSEMBLY/ASSEMBLY cont.

Disconnect the probe cable connector at the readout assembly. Disassemble the probe by first removing the exhaust screw at the base of the probe adjacent to the handle (see Figure 5-1). Grasp the end cap in one hand and the probe shell in the other, gently pull to separate the end cap and the lamp housing from the shell.

Hold the lamp housing with the black end cap upright. Loosen the screws on the top of the end cap, separate the end cap and ion chamber from the lamp and lamp housing.

CAUTION

Care must be taken so that the ion chamber does not fall out of the end cap or the light source does not fall out of the lamp housing.

Turn the end cap over in the hand. Tap lightly on the top. The ion chamber should fall out of the end cap into the hand.

Place one hand over the top of the lamp housing and tilt slightly. The light source will slide out of the housing.

The amplifier board can be removed from the lamp source housing assembly (see Figure 5-2) by unsnapping the coaxial connector, J1, and then removing the retaining screw. The amplifier board will then slide out of the housing assembly.

Reassemble the probe by first sliding the lamp back into the lamp housing. Place the ion chamber on top of the lamp housing, making sure that the contacts are properly aligned. The ion chamber fits only one way.

If the ion chamber is to be replaced always use one identical to the one being removed. Check the aperture (small: 3.0 mm; large: 6.0 mm) at the top of the ion chamber and materials of construction (gold-plated or Teflon) to ensure proper replacement. See Parts List, Section 7.

Place the end cap on top of the ion chamber and replace the two screws. Tighten the screws only enough to seal the O-ring.

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NHN PHOTOIONIZATION DETECTOR MAINTENANCE

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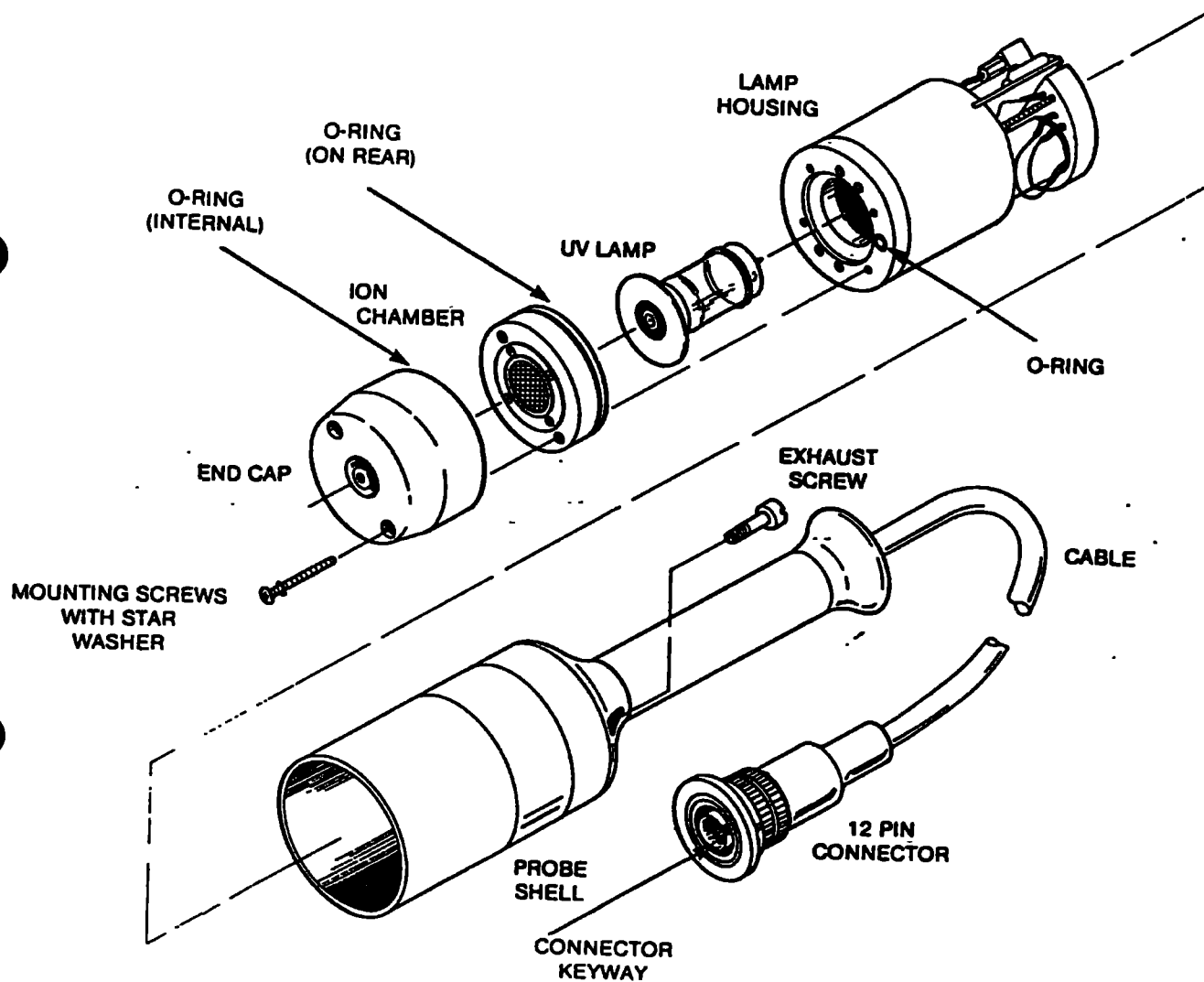


FIGURE 1
PROBE ASSEMBLY

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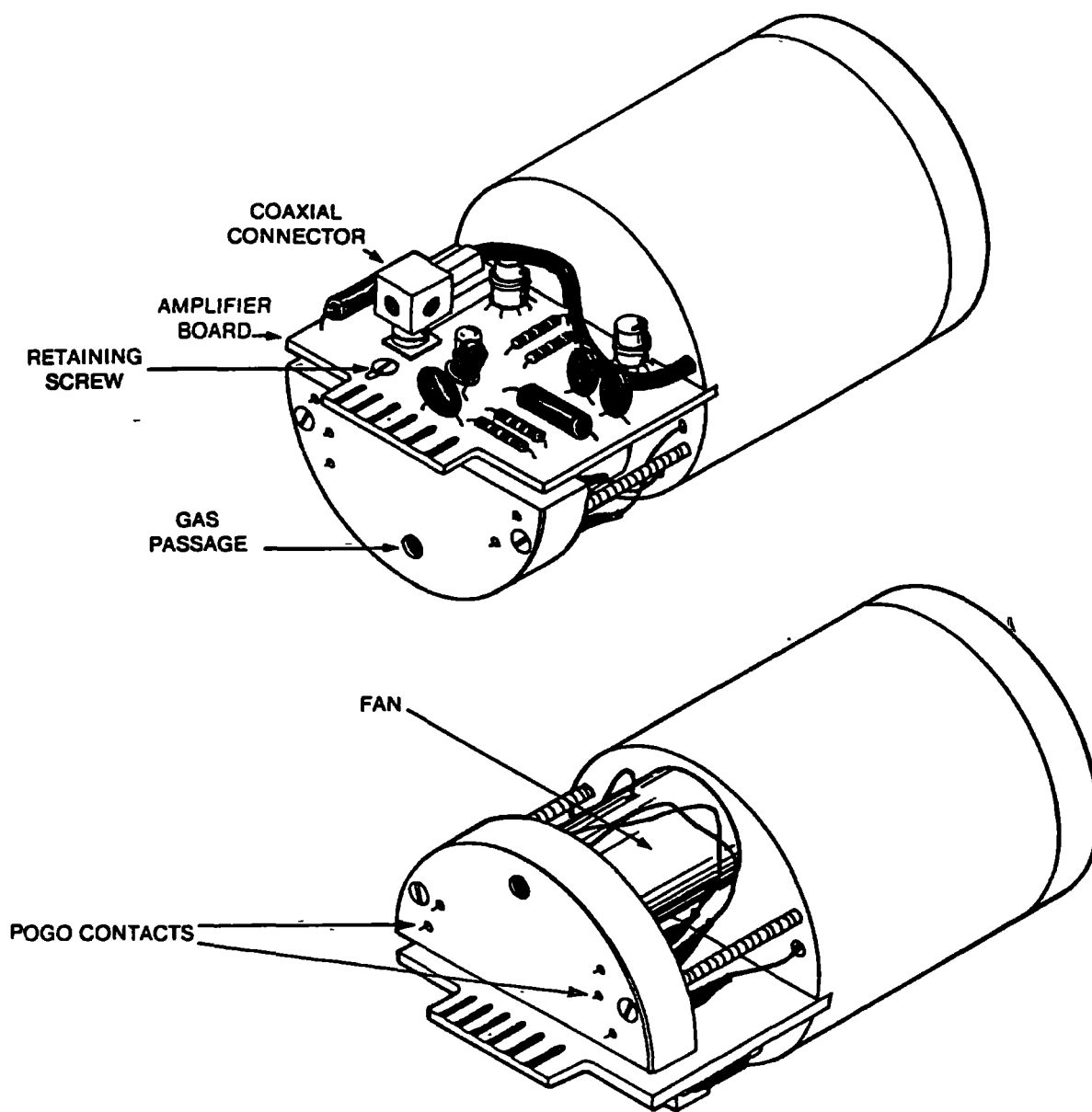


FIGURE 2
LAMP HOUSING ASSEMBLY

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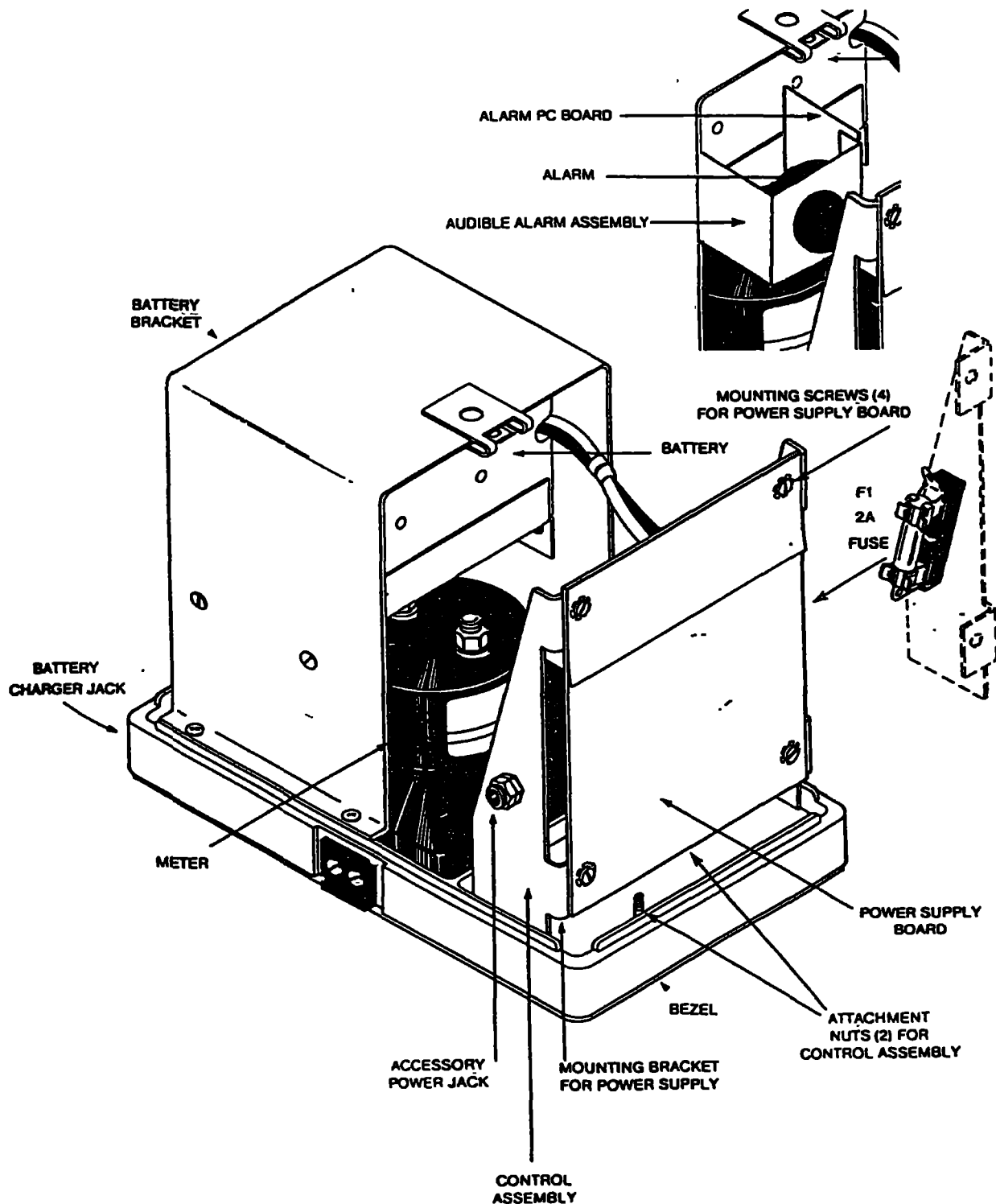


FIGURE 3
READOUT ASSEMBLY

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SECTION 5.5, PROBE DISASSEMBLY/ASSEMBLY cont.

CAUTION

Do not over-tighten these screws.

Line up the pins (pogo contacts) on the base of the lamp housing with the pins inside the probe shell. Gently slide the housing assembly into the probe shell.

The end cap should meet the probe shell evenly after final assembly. If not, the ion chamber may be installed wrong.

CAUTION

DO NOT FORCE the assembly into the shell.
It fits only one way.

If it does not reassemble readily, remove and check pin alignment. Check to ensure pogo contacts are not bent. Refasten the exhaust screw at the base of the probe.

Align the 12 pin probe connector to the readout assembly and reconnect with a twisting motion until a click occurs. Check to ensure the high voltage microswitch is properly depressed. The lamp should light if the function switch is turned to any position except **STANDBY**.

5.6 READOUT DISASSEMBLY/ASSEMBLY

WARNING

Turn the function switch on the control panel to the **OFF** position before disassembly. Otherwise, high voltage of 1200 V DC will be present.

Disconnect the probe cable connection. Remove recorder jacks and cable or the plastic plug cap. Loosen the screw on the bottom of the case and, holding the instrument by the bezel, remove the case (see Figure 5-3).

- a. The control assembly consisting of the Printed Circuit Board (PCB) and control panel can be separated from the readout assembly by the following steps:

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NHN PHOTOIONIZATION DETECTOR MAINTENANCE

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SECTION 5.6, READOUT DISASSEMBLY/ASSEMBLY cont.

- 1) Separate the Molex connectors in the cables to the control assembly.
- 2) Remove the two attachment nuts at the base of the assembly.
- 3) Remove the two screws at the top of the power supply board holding it to the assembly brackets.
- 4) Compress the brackets and slide the assembly thru the bezel. Remove a third screw at the lower corner of the board, if necessary.

b. The optional alarm assembly can be separated as follows

- 1) Disconnect the cable (P6/J6 of Figure 4-5)
- 2) Remove the two screws holding the alarm assembly to the battery bracket

Reassembly is accomplished by reversing the above procedure.

NOTE: Be sure the function switch on the control panel is in the OFF position before inserting the control module into the case. If not, the fuse can be blown or damage can result.